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(54) Title: GRANULATED FLAVOR AND METHOD FOR PRODUCING THE SAME

(57) Abstract: The present invention provides a method for producing a granulated flavor, comprising: forcing a powdered flavor prepared from raw materials containing a flavor and a carrier to free-fall; conveying the powdered flavor with a horizontal screw to obtain a pressed powdered flavor; compacting the pressed powdered flavor with rollers to obtain a partially melted plated matter; cooling the plated matter; and grinding and granulating the cooled plated matter. According to the present invention, a granulated flavor is provided which releases flavor immediately when needed while the storage stability of the aroma is superior. The granulated flavor of the invention is less fragile and thus less likely to crumble upon transportation of the granulated flavor itself or of the food or beverage containing the granulated flavor. The granulated flavor as prepared above has a moisture content of 10% or less by weight and a hardness of 1 N/mm²-50 N/mm².

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DESCRIPTION

GRANULATED FLAVOR AND METHOD FOR PRODUCING THE SAME

5 Technical Field

The present invention relates to a method for producing a granulated flavor and a granulated flavor for use in foods and beverages with specific moisture content and hardness. More particularly, the present invention relates to a granulated
10 flavor for use in foods and beverages (hereinafter, sometimes referred to as a "granulated flavor") with outstanding storage stability whose form is less likely to crumble and which releases flavor immediately when needed.

15 Background Art

Examples of conventionally known methods for powdering flavors include spray drying, freeze drying and absorption process. When a powdered flavor is produced by spray drying, the resulting powder has a relatively small size of 50 to 200
20 μm , which not only has a poor flow property but also has a disadvantage of a poor solubility in cold water and the like. In this respect, freeze drying in which raw materials are frozen and dried resolves the above-described disadvantages of spray drying. However, since freeze drying requires longer time in
25 drying, it is less efficient. Absorption process requires shorter processing time since powdered flavor can be obtained through a simple manipulation of mixing liquid and powder, and

thus seems a rational process. However, the resulting powder has problems of being susceptible to flavor deterioration upon storage and of being highly hygroscopic.

On the other hand, since a conventional granulation process is known to result in flavor deterioration due to the heating process for evaporating water, a technique in which fatty oil and powdered food are mixed together and then the mixture is subjected to dry compaction granulation is reported (e.g., see Patent document 1: Japanese Patent Laid-Open Application No. 11-276144 (especially, 2nd col., lines 8-11)).

Furthermore, since a conventional granulation process is known to lose aroma during the heating step and thus results in flavor deterioration, a method of compaction granulation without adding water, in which powder ingredient with 10% or greater extracted powdered flavor component is used while the moisture content in the powder ingredient is 1-5% is reported (e.g., see Patent document 2: Japanese Patent Laid-Open Application No. 11-239461 (especially, 3rd col., lines 16-18)).

During having studied a powdered flavor obtained through the above-described dry compaction granulation, the present inventors blended the powdered flavor in a chewing-gum base to obtain a stick gum and they found that when chewing this stick gum the release of the flavor tends to be late and that the aroma is weakened. Through diverse studies, the present inventors assumed that this phenomenon is due to the breakdown of the powdered flavors by which the flavor inside the powder is released and dissolved in the chewing-gum base. Thus, the objectives

of the present invention is to provide a granulated flavor which is less likely to crumble, and to provide a granulated flavor having the above-described property as well as a good aroma storage stability and immediate release of flavor when needed.

5

Disclosure of Invention

The present inventors have devoted themselves to studies to find out solution to the above-described objectives. As a result, they found that when a partially-melted plated matter
10 obtained by firmly compacting a conventional powdered flavor with rollers is cooled down and then subjected to grinding and granulation, a granulated flavor which is less likely to breakdown and which has a good aroma storage stability and immediate release of flavor when needed. The present inventors
15 continued further studies and found that by forcing a powdered flavor prepared from raw materials containing a flavor and a carrier to free-fall into the feed hopper, conveying it with a horizontal screw to obtain a pressed powdered flavor, compacting the pressed powdered flavor with rollers to obtain
20 a plated matter, cooling down the partially-melted plated matter and subjecting the cooled plated matter to grinding and granulation, the resulting granulated flavor is difficult to crumble, has a good aroma storage stability and immediately releases flavor when needed, thereby achieving the present
25 invention.

Thus, the present invention provides the followings.

- A method for producing a granulated flavor, comprising:

compacting a powdered flavor prepared from raw materials containing a flavor and a carrier with rollers to obtain a partially melted plated matter; cooling the plated matter; and grinding and granulating the cooled plated matter.

5 - A method for producing a granulated flavor, comprising: forcing a powdered flavor prepared from raw materials containing a flavor and a carrier to free-fall into the feed hopper; conveying the powdered flavor with a horizontal screw to obtain a pressed powdered flavor; compacting the pressed powdered flavor with
10 rollers to obtain a partially melted plated matter; cooling the plated matter; and grinding and granulating the cooled plated matter.

 - A granulated flavor having a moisture content of 10% or less by weight and a hardness of 1 N/mm^2 - 50 N/mm^2 .

15 - The above-described granulated flavor containing particles having a particle size of $105 \mu\text{m}$ - 2 mm , wherein the proportion of the particles having that particle size is 85% or more by weight.

 - A food or beverage comprising the above-described
20 granulated flavor.

Best Modes for Carrying Out the Invention

Hereinafter, the present invention will be described in more detail.

25 First, flavor as one of the raw materials for composing a granulated flavor will be described. The flavor used in the present invention is not particularly limited as long as it is

capable of achieving the above-described objectives. Examples of flavors from natural sources include: an essential oil obtained through squeezing or steam distillation of plant materials; oleoresin obtained by extracting a plant material with a carbon-dioxide-containing solvent, filtering off insoluble matter and removing the solvent; a flavor as an oil or a concentrated aqueous solution collected by concentrating juice while introducing aroma component that effuses with water into a collector; an extract obtained by contacting a plant or animal material with various solvents to extract a necessary flavor component from the material, remove these solvent if necessary and concentrate the flavor component; an isolated flavor by purifying a compound from a mixture; a cooked flavor produced by heating a food material; an enzyme modified flavor produced through biochemical reaction using dairy material, protein or fat as a substrate. Examples of other flavors include chemically synthesized flavors such as amyl acetate, benzyl alcohol, geraniol and phenylethyl alcohol. Examples of natural flavor sources include dried bonito extract, tangle weed extract, crab extract, oyster extract, beef extract, chicken extract, pork extract, onion extract, carrot extract, orange juice, lemon juice, grapefruit juice and the like. These sources may be used alone or two or more of these sources may be used in combination. Any blended flavor obtained by suitably selecting and mixing these sources may be used.

Preferable flavors include, flavors such as essential oils and oleoresins, synthetic flavors such as 1-menthol, citral,

geraniol and vanillin, citrus flavors such as orange, lemon and grapefruit, fruit flavors such as apple, banana, grape, peach, strawberry, melon and pineapple, mint flavors such as peppermint and spearmint, spice flavors such as pepper, cinnamon, nutmeg and clove, nut flavors such as vanilla, coffee, cocoa and hazelnut, cooked or uncooked meat/seafood flavors such as beef, pork, chicken, salmon, crab and shrimp, tea flavors such as black tea, green tea and oolong tea, and dairy flavors such as milk, cheese and butter.

Next, carriers that partially consist the granulated flavor will be described.

Carriers are preferably hydrophilic, including hydrophilic proteins such as gelatin, casein, casein sodium, whey protein, powdered skimmed milk, powdered whole milk, albumin; hydrophilic polysaccharides such as maltodextrin, modified starch (acid digested starch, oxidized starch, pregelatinized starch, grafted starch, etherified starch, esterified starch obtained by reacting with acetic acid, phosphoric acid or the like), alginate, acacia gum, soybean polysaccharide, guar gum, xanthan gum, pectin, carboxymethyl cellulose and agarose; partially hydrolyzed proteins such as HAP and HVP; partially decomposed starches such as oligosaccharide; and saccharides such as lactose. In addition to these substance, a carrier may also contain at least one of organic acids such as acetic acid and citric acid, salts used for general food such as sodium chloride and potassium chloride, and the like.

More specifically, examples include monosaccharides such as glucose and fructose, oligosaccharides such as sucrose, maltose and trehalose, polysaccharides such as starch and modified starch, and proteins such as casein, gelatin and whey protein.

A powdered flavor used as a raw material in the present invention is those that are prepared by known process. Specifically, a powdered flavor obtained through a known methods such as spray drying and absorption process can be used as the raw material.

Spray drying for producing a powdered flavor can be any methods known prior to the present application. Although a typical process is described herein, the present invention is not limited to this process. Specifically, first, water, a carrier selected from the above-mentioned carriers and, if necessary, an emulsifier are mixed, heated and sterilized. Then, a pre-weighed flavor is added, agitated and mixed using a mixer such as a TK mixer to emulsify the mixture. The obtained emulsion is spray dried with a spray drier followed by sieving to obtain the powdered flavor.

Absorption process for producing a powdered flavor can be any methods known prior to the present application. A typical process includes mixing and absorbing a carrier selected from the above-mentioned carriers with a pre-weighed flavor to obtain a powdered flavor.

The above-described powdered flavor is compacted with rollers. In general, the powdered flavor is fed between the

rollers for compaction. The means for feeding the powdered flavor is not particularly limited. For example, a horizontal screw is preferably used to feed the powdered flavor. The powdered flavor fed between the rollers is firmly compacted by the rotation of the rollers. As the rollers continue to rotate, a plated matter comes out from the gap between the rollers. The plated matter needs to be firmly compacted such that it is partially in a melted form. Herein, the plated matter refers to a wide zone with a constant thickness or an equivalent thereof.

10 A "partially melted plated matter" refers to a plated matter that is not totally melted so that it can still hold itself. In other words, the surface of the plated matter is partially or fully in a viscous state while the inside is hard enough to support itself so that there is no need of depending on use of other support to proceed to the next step.

In order to obtain the above-described plated matter in a partially melting state, it is requisite to firmly compact the powdered flavor. Although it depends on the materials used for the powdered flavor, the roller compression pressure on the powdered flavor is generally 15 N/cm or greater, or even preferably 20 N/cm or greater to obtain favorable results. In the specification, the word "compress" has a broad meaning such that it includes the word "compact". The upper limit of the roller compression pressure is, although depending on the compressor used for the powdered flavor, for example, about 60 N/cm. The term "roller compression pressure" refers to the performance of the rollers, that is, a compressive load per unit

compression width upon compressing the powdered flavor with the rollers.

The roller compression pressure (N/cm) may be calculated by dividing the force put upon the rollers (N) with the effective
5 length of the rollers (cm) (the length of the rollers that makes contact with and compacts the powdered flavor), where the force put upon the roller (N) is the product of the hydraulic pressure (gauge pressure) of the rollers (N/cm²) and the cross-sectional area (cm²) of the supporting rod.

10 According to the present invention, preferable conditions for compacting the powdered flavor depend on the materials used for the powdered flavor to be compacted, the amount of the powdered flavor fed between the rollers, the gap between the rollers, the compression pressure of the rollers and the revolution of
15 the rollers. For example, the revolution of the screw for feeding the powdered flavor between the rollers may be 10-20 rpm while the compression pressure of the rollers may be 15 N/cm or greater, and more favorable results can be obtained when the compression pressure of the rollers is 20 N/cm or greater and the revolution
20 of the rollers is 12-20 rpm.

Then, the partially melted plated matter is cooled down. Ambient air around the plated matter may be utilized to cool down the plated matter but preferably the plated matter is actively cooled down by being blown with air or cooled air
25 utilizing a fan or the like. Subsequently, the cooled plated matter is ground and granulated. Any method may be used as long as the above-described objectives are achieved.

According to the present invention, the powdered flavor is preferably pressed prior to the firm compaction process with the rollers. Specifically, the powdered flavor is preferably forced to free-fall by its own weight and then conveyed with the horizontal screw, thereby pressing and preferably deaerating the powdered flavor. Deaerating is preferably performed in the step of conveying the flavor with the horizontal screw. When deaeration is done in this step, the following process can proceed smoothly.

Thus, the granulated flavor of the invention is prepared. The granulated flavor of the invention can be used if the moisture content is 15% or less by weight, preferably 10% or less by weight.

When the granulated flavor of the invention with a high moisture content is blended with a product that tends to uptake moisture and added with a considerable amount of amino acids, carbohydrate and organic acid, such as a powdered beverage (powdered tea, powdered juice, powdered soup) or a powdered seasoning (powdered chemical seasoning, powdered broth, powdered soybean sauce, powdered sauce), the whole product cakes, rendering itself commercially unworthy. Therefore, it is necessary to control the moisture content of the granulated matter to be added. Although the lower moisture content is the better, the minimum limit is practically about 0.5% by weight.

The moisture content may be determined using a known method. As common methods, Karl Fischer methods are known, which are detailed in the Japanese Pharmacopoeia. According to the present invention, a Karl Fischer moisture meter (Kyoto

Electronics Manufacturing Co. Ltd.: MKA-210) is used to measure the moisture according to the Japanese Pharmacopoeia.

The granulated flavor obtained by the invention gives favorable results when its hardness is 1 N/mm^2 - 50 N/mm^2 . If the hardness is lower than 1 N/mm^2 , the granulated flavor is likely to crumble and be fragile to external force, and thus difficult to maintain the form of the granule. If the hardness is greater than 50 N/mm^2 , the solubility of the obtained granulated flavor becomes low, which not only prevents a high flavor content but also increases production cost. Regarding the flavor content and production cost, the hardness of the granulated flavor is preferably about 1 N/mm^2 - about 10 N/mm^2 , and more preferably about 1 N/mm^2 - about 5 N/mm^2 .

According to the present invention, the hardness can be measured by a known method. Specifically, a particle hardness tester Model Type GM (Okada Seiko Co., Ltd.) was used. A granulated flavor whose hardness is to be measured is selected, put under an increasing load at a constant speed and calculate the hardness based on the load at which the form of the granulated flavor breaks down. Although the granulated flavor of the invention is very hard, it is soluble and thus rapidly dissolves in the presence of moisture.

Preferably, the granulated flavor of the invention contains particles with a size of $105 \mu\text{m}$ - 2 mm for 85% or more by weight. More preferably, the granulated flavor of the invention contains particles with a size of $105 \mu\text{m}$ - 2 mm for 90% or more by weight. However, preferable particle sizes depend

on the usage of the granulated flavor of the invention. For example, in the case of a powdered beverage whose major ingredients are granulated sugar, citric acid and a powdered flavor, when the difference among the particle sizes of these ingredients is great, they may cause undesirable classification resulting in a problem of unevenness. Therefore, a granulated flavor having a particle size closer to that of the granulated sugar is required, in this case the granulated flavor with a particle size of preferably 2.0 mm-500 μ m, more preferably 2 mm-1 mm is used. In the case of a powdered seasoning that is separately packed and used with an instant noodle, which contains chemical seasonings, meat extract powder, vegetable powder and the like, the granulated flavor with a particle size of preferably 1 mm-105 μ m, more preferably 500 μ m-105 μ m is used.

Particle size distribution is measured using a JIS standard sieve and separated by particle sizes of 2.0 mm or larger, 2.0-1.0 mm, 1.0 mm-500 μ m, 500 μ m-250 μ m, 250 μ m-105 μ m and 105 μ m or smaller.

Since the granulated flavor obtained according to the present invention is provided, for example, in a soft capsule, a paper tube or a soup package attached with an instant noodle, it is beneficial that the relative density of the powder is as high as possible for compact packaging. Specifically, the apparent density of the granulated flavor obtained by the invention is preferably 1 g/cm³-0.81 g/cm³. The method for determining the apparent density is known. According to the present invention, a powder tester (Hosokawa Micron Corporation)

is used to determine the apparent density (g/cm²).

Examples of food products that can be blended with the granulated flavor obtained by the invention include, but not limited to, beverages, powdered beverages, confectionery products such as candy, chewing-gum, tablet candy, chocolate and snacks, desserts such as yogurt and ice-cream, meat/seafood products, powdered seasonings, food such as powdered food (e.g., fillings for rice in hot tea) and precooked products to be heated in a microwave oven (frozen food, pouch-packed food). An amount of the granulated flavor of the invention in a product is not particularly limited and depends on the product and the granulated flavor used.

EXAMPLES

Hereinafter, the present invention will be described more specifically by way of Examples, Comparative Examples, Application Examples and Test Examples. However, the present invention is not in any way limited to these examples.

Example 1

Ten kg of trehalose (Hayashibara Shoji, Inc.) was blended with 100 g of color materials agents (Yellow No.4) and then mixed and absorbed with 0.2 kg of lemon flavor (Takasago International Corporation), thereby obtaining an absorbed powdered flavor. This absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 15 rpm to be fed between the compression rollers. The powdered flavor was compacted at a roller compression

pressure of 25 N/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was cooled with cold air. The cooled plated matter was ground with a rotary grinding machine and evened the size, thereby obtaining a
5 granulated matter.

Comparative Example 1

Ten kg of trehalose (Hayashibara Shoji, Inc.) was blended with 100 g of color materials agents (Yellow No.4) and then mixed and absorbed with 0.2 kg of lemon flavor (Takasago International
10 Corporation), thereby obtaining an absorbed powdered flavor.

Comparative Example 2

Ten kg of trehalose (Hayashibara Shoji, Inc.) was blended with 100 g of color materials agents (Yellow No.4) and then mixed and absorbed with 0.2 kg of lemon flavor (Takasago International
15 Corporation), thereby obtaining an absorbed powdered flavor. This absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 48 rpm to be fed between the compression rollers. The powdered flavor was compressed at a roller compression
20 pressure of 7 N/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was ground without cooling with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

Example 2

25 Ten kg of anhydrous crystalline maltose (Hayashibara Shoji, Inc.) was mixed and absorbed with 1 kg of menthol flavor (Takasago International Corporation), thereby obtaining an absorbed

powdered flavor. This absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 15 rpm to be fed between the compression rollers. The powdered flavor was compacted at a roller compression pressure of 25 N/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was cooled with cold air. The cooled plated matter was ground with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

10 Comparative Example 3

Ten kg of anhydrous crystalline maltose (Hayashibara Shoji, Inc.) was mixed and absorbed with 1 kg of menthol flavor (Takasago International Corporation), thereby obtaining an absorbed powdered flavor.

15 Comparative Example 4

Ten kg of anhydrous crystalline maltose (Hayashibara Shoji, Inc.) was mixed and absorbed with 1 kg of menthol flavor (Takasago International Corporation), thereby obtaining an absorbed powdered flavor. This absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 48 rpm to be fed between the compression rollers. The powdered flavor was compressed at a roller compression pressure of 7 N/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was ground without cooling with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

Example 3

Ten kg of anhydrous crystalline maltose (Hayashibara Shoji, Inc.) was mixed and absorbed with 0.5 kg of lemon flavor (Takasago International Corporation), thereby obtaining an absorbed
5 powdered flavor. This absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 15 rpm to be fed between the compression rollers. The powdered flavor was compacted at a roller compression pressure of 25 N/cm, a roller gap of 0.5
10 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was cooled with cold air. The cooled plated matter was ground with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

Comparable Example 5

15 Ten kg of anhydrous crystalline maltose (Hayashibara Shoji, Inc.) was mixed and absorbed with 0.5 kg of lemon flavor (Takasago International Corporation), thereby obtaining an absorbed powdered flavor.

Comparable Example 6

20 Ten kg of anhydrous crystalline maltose (Hayashibara Shoji, Inc.) was mixed and absorbed with 0.5 kg of lemon flavor (Takasago International Corporation), thereby obtaining an absorbed powdered flavor. This absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a
25 horizontal screw at a screw revolution of 48 rpm to be fed between the compression rollers. The powdered flavor was compressed at a roller compression pressure of 7 N/cm, a roller gap of 0.5

mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was ground without cooling with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

5 Example 4

Ten kg of anhydrous crystalline maltose (Hayashibara Shoji, Inc.) was mixed and absorbed with 0.2 kg of apple flavor (Takasago International Corporation), thereby obtaining an absorbed powdered flavor. This absorbed powdered flavor was forced to
10 free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 15 rpm to be fed between the compression rollers. The powdered flavor was compacted at a roller compression pressure of 25 N/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter.
15 The plated matter was cooled with cold air. The cooled plated matter was ground with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

Comparative Example 7

Ten kg of anhydrous crystalline maltose (Hayashibara Shoji, Inc.) was mixed and absorbed with 0.2 kg of apple flavor (Takasago International Corporation), thereby obtaining an absorbed powdered flavor.
20

Comparative Example 8

Ten kg of anhydrous crystalline maltose (Hayashibara Shoji, Inc.) was mixed and absorbed with 0.2 kg of apple flavor (Takasago International Corporation), thereby obtaining an absorbed powdered flavor. This absorbed powdered flavor was forced to
25

free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 48 rpm to be fed between the compression rollers. The powdered flavor was compressed at a roller compression pressure of 7 N/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was ground without cooling with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

Example 5

10 Ten kg of sorbitol (Merck Ltd., Japan) was mixed and absorbed with 0.2 kg of apple flavor (Takasago International Corporation), thereby obtaining an absorbed powdered flavor. This absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw
15 revolution of 15 rpm to be fed between the compression rollers. The powdered flavor was compacted at a roller compression pressure of 25 N/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was cooled with cold air. The cooled plated matter was ground with a rotary
20 grinding machine and evened the size, thereby obtaining a granulated matter.

Comparative Example 9

Ten kg of sorbitol (Merck Ltd., Japan) was mixed and absorbed with 0.2 kg of apple flavor (Takasago International
25 Corporation), thereby obtaining an absorbed powdered flavor.

Comparative Example 10

Ten kg of sorbitol (Merck Ltd., Japan) was mixed and

absorbed with 0.2 kg of apple flavor (Takasago International Corporation), thereby obtaining an absorbed powdered flavor. This absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw
5 revolution of 48 rpm to be fed between the compression rollers. The powdered flavor was compressed at a roller compression pressure of 7 N/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was ground
10 without cooling with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

The powders obtained as described above were evaluated as follows.

Application Example 1: Production of chewing-gums
containing the granulated flavors

15 To 100 g of chewing-gum base (Takasago International Corporation), 5 g of the powdered flavor obtained in Comparable Example 1, and 5 g each of the granulated flavors obtained in Comparable Example 2 and Example 1 were blended and mixed at about 50°C, respectively. After cooling, they were roll-molded
20 with rollers, thereby obtaining three types of 3 g stick gums.

Test Example 1: Examination of the form of the granulated
flavors

The stick gums obtained in Application Example 1 were examined. The granulated flavor obtained in Example 1 generally
25 maintained its form in the stick gum. On the other hand, considerable amounts of the powdered flavor from Comparative Example 1 and the granulated flavor from Comparative Example

2 lost their forms in the stick gums.

Test Example 2: Sensory evaluation of the granulated flavors

The stick gums obtained in Application Example 1 were
 5 subjected to sensory evaluation on the following basis. Ten
 specialized panelists participated in the sensory evaluation
 to evaluate aroma and taste of the stick gums, which were imparted
 by the granulated flavors added therein. The intensity of the
 flavor release and the total judgment of the taste of the stick
 10 gums were made at 15 seconds, 30 seconds, 1 minute and every
 1 minute until 5 minutes after the beginning of chewing the stick
 gums. The intensity of the flavor release was evaluated on the
 grade of four scale (i.e., weak, rather weak, rather strong,
 strong) while the taste was evaluated as tasty or not tasty.
 15 The results of the evaluation are shown in Table 1.

Table 1

Granulated flavor	Total judgment	Release (Intensity of the flavor released from the gum base)		
		Early	Middle	Later
Comparative Example 1	Not tasty (10) Tasty (0)	Weak	Rather strong	Rather strong
Comparative Example 2	Not tasty (7) Tasty (3)	Rather weak	Rather strong	Rather strong
Example 1	Not tasty (1) Tasty (9)	Strong	Rather strong	Rather weak

In Table 1, "Early" means 0-1 minute, "Middle" means from
 20 after 1 minute till 3 minutes and "Later" means from after 3

minutes till 5 minutes after the beginning of chewing.

From the results of the total judgment of tastes shown in Table 1, the stick gum containing the granulated flavor obtained in Example 1 was sensed tasty. Presumed reasons for this result is that the stick gum containing the granulated flavor obtained in Example 1 releases the lemon freshness flavor immediately after the beginning of chewing while the stick gums containing the powdered flavor obtained in Comparative Example 1 and the granulated flavor obtained in Comparative Example 2 take time to release the flavors and the intensities of their releases are weaker. Presumably, as observed in previous Test Example 1, since the powders from Example 1 remain their forms in the stick gum, the flavor is retained in the granulated flavor, and thus the flavor release is effective upon chewing the stick gum.

Test Example 3: Fragility Endurance test of the granulated flavors

The granulated flavors obtained in Comparative Examples and Examples shown in Table 2 were put in tubular containers (diameter: 6 cm, height: 10 cm) for 50 g, respectively. The containers were shaken with a reciprocating vibrator at a stroke of 4 cm and a vibration frequency of 160/min. for 120 minutes. The particle sizes before and after the vibration were measured using a JIS standard sieve to determine the fragility of the granulated flavors. The results are shown in Table 2.

Table 2

Granulated flavor	Particle size before vibration	Particle size after vibration
Comparative Example 4	1.7 mm-1mm products	1 mm or smaller products 32.7%
Example 2	1.7 mm-1mm products	1 mm or smaller products 17.5%
Comparative Example 2	1.7 mm-1mm products	1 mm or smaller products 10.0%
Example 1	1.7 mm-1mm products	1 mm or smaller products 6.0%

Test Example 4: Storage stability of the granulated flavors

The powdered flavor obtained in Comparative Example 1 and the granulated flavors obtained in Comparative Example 2 and Example 1 were stored in a thermostatic chamber at 60°C. At one week and two weeks after the storage, the aromas of the granulated flavors were evaluated by the specialized panelists. The results of the evaluation are shown in Table 3.

10

Table 3

	At one week	At two weeks
Comparative Example 1	Great change (10 panelists)	Great change (aroma faded away/weak) (10 panelists)
Comparative Example 2	Some change (9 panelists)	Recognize oxidization deterioration (9 panelists)
Example 1	No change (10 panelists)	Almost no change (9 panelists)

Referring to Table 3, the powdered flavor from Comparative Example 1 almost lost its aroma in the powdered flavor after

2 weeks and thus became worthless as a product. The granulated flavor from Comparative Example 2 was inferior to the granulated flavor from Example 1 in stability. This proves that the compression step at high pressure in the production process of the granulated flavor deaerates and densifies the matrix of the granulated flavor, which prevents air oxidation and aroma lost.

Test Example 5: Hardness of the granulated flavors

The granulated flavors obtained in Comparative Examples and Examples indicated in Table 4 were placed on a sample table and subjected to increasing load at a speed of 100 $\mu\text{m}/\text{sec}$ to determine the load peak values at which the granulated flavors lose their forms. The values were applied into a certain formula $4P/(\pi \times D \times D)$ to calculate the hardness of the granulated flavors. In the formula, P represents the load peak value and D represents the particle size of the granulated flavor. The results are shown in Table 4.

Table 4

Granulated flavor	Hardness mN/mm^2
Example 1	1992
Comparative Example 2	176
Example 2	1293
Comparative Example 4	644
Example 3	1358
Comparative Example 6	801
Example 4	4885
Comparative Example 8	882

Test Example 6: Distribution of particle sizes of the granulated flavors

The distributions of the particle sizes of the granulated flavors prepared in Examples and Comparative Examples indicated in Table 5 were determined using a JIS standard sieve. The results are shown in Table 5.

5

Table 5

Granulated flavor	2 mm or larger	2mm to 1mm	1mm to 500 μ m	500 μ m to 250 μ m	250 μ m to 105 μ m	105 μ m or smaller
Example 1	0.9	67.6	16.9	6.8	4.9	2.9
Comparative Example 4	10.7	4.9	7.6	11.3	53.5	12.0
Example 5	3.5	54.5	19.8	7.9	7.0	7.3
Comparative Example 10	1.4	39.2	18.8	6.1	11.7	22.8

From these results, the proportions of small particle sizes of the granulated flavors of the invention are less than those of the granulated flavors from the Comparative Examples. This proves that the granulated flavors of the invention have a higher granulation yield and thus useful in terms of industrial use.

Example 6

First, 15 kg of water, 8.9 kg of dextrin (Matsutani Chemical Industry Co., Ltd.) as a carrier and 0.1 kg of emulsifier (polyglycerine fatty acid ester) were dissolved, to which 1 kg of lemon oil (Takasago International Corporation) was added as a flavor. A TK mixer was used for agitation and mixing to obtain an emulsion. The emulsion was spray dried with a spray drier (inlet temperature: 180°C, outlet temperature: 90°C) to obtain a powdered flavor. The powdered flavor was forced to free-fall

in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 15 rpm to be fed between the compression rollers. The powdered flavor was compacted at a roller compression pressure of 25 N/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was cooled with cold air. The cooled plated matter was ground with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

Comparative Example 11

First, 15 kg of water, 8.9 kg of dextrin as a carrier and 0.1 kg of emulsifier (polyglycerine fatty acid ester) were dissolved, to which 1 kg of lemon oil (Takasago International Corporation) was added as a flavor. A TK mixer was used for agitation and mixing to obtain an emulsion. The emulsion was spray dried with a spray drier (inlet temperature: 180°C, outlet temperature: 90°C) to obtain a powdered flavor. The powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 48 rpm to be fed between the compression rollers. The powdered flavor was compacted at a roller compression pressure of 7 ton/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was ground without cooling with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

Example 7

To 10 kg of powdered skimmed milk (Meiji Dairies Corporation), 0.2 kg of milk flavor (Takasago International

Corporation) was pre-absorbed to obtain an absorbed powdered flavor. This absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 15 rpm to be fed between the compression rollers. The powdered flavor was compacted at a roller compression pressure of 25 N/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was cooled with cold air. The cooled plated matter was ground with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

Example 8

After mixing 4.9 kg of acacia gum (Takasago International Corporation), 4.9 kg of dextrin (Matsutani Chemical Industry Co., Ltd.) and 0.2 kg of gelatin (Nitta Gelatin Inc.), 0.2 kg of pepper mint flavor (Takasago International Corporation) was added and mixed to absorb, thereby obtaining an absorbed powdered flavor. This absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 15 rpm to be fed between the compression rollers. The powdered flavor was compacted at a roller compression pressure of 25 N/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was cooled with cold air. The cooled plated matter was ground with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

Example 9

After mixing 7.0 kg of dextrin (Takasago International

Corporation), 2.8 kg of modified starch (Matsutani Chemical Industry Co., Ltd.) and 0.2 kg of carboxymethyl cellulose (Shin-Etsu Chemical Co., Ltd.), 0.2 kg of coffee flavor (Takasago International Corporation) was added and mixed to absorb, thereby obtaining an absorbed powdered flavor. This absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 15 rpm to be fed between the compression rollers. The powdered flavor was compacted at a roller compression pressure of 25 N/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was cooled with cold air. The cooled plated matter was ground with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

The granulated coffee flavor obtained in Example 9 was used to prepare a coffee soft candy having the following composition.

Ingredients	Composing amount (part weight)
Granulated coffee flavor	0.3
Starch syrup	900
Granulated sugar	600
Lactose	100
hydrogenated vegetable oil	100
Fondant	100
Gelatin	10
Lecithin	1

After mixing 9.8 kg of dextrin (Matsutani Chemical Industry Co., Ltd.) and 0.2 kg of casein (Taiyo Kagaku Co., Ltd.), 0.2 kg of black tea flavor (Takasago International Corporation) was added and mixed to absorb, thereby obtaining an absorbed powdered
5 flavor. This absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 15 rpm to be fed between the compression rollers. The powdered flavor was compacted at a roller
compression pressure of 25 N/cm, a roller gap of 0.5 mm and a
10 roller revolution of 15 rpm to obtain a plated matter. The plated matter was cooled with cold air. The cooled plated matter was ground with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

Example 11

15 After mixing 5.0 kg of powdered skimmed milk (Morinaga Milk Industry Co., Ltd.) and 5.0 kg of powdered whole milk (Morinaga Milk Industry Co., Ltd.), 0.2 kg of butter flavor (Takasago International Corporation) was added and mixed to absorb, thereby obtaining an absorbed powdered flavor. This
20 absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 15 rpm to be fed between the compression rollers. The powdered flavor was compacted at a roller compression pressure of 25 N/cm, a roller gap of 0.5 mm and a roller revolution
25 of 15 rpm to obtain a plated matter. The plated matter was cooled with cold air. The cooled plated matter was ground with a rotary grinding machine and evened the size, thereby obtaining a

granulated matter.

The granulated butter flavor obtained in Example 11 was used to prepare a butter bread having the following composition.

Ingredients	Composing amount (part weight)
Granulated butter flavor	0.1
Strong flour	100
Shortening	5
Sugar	5
Wet yeast	2.5
Salt	2.1
Powdered skimmed milk	2
Water	67

5

Example 12

To 10.0 kg of trehalose (Hayashibara Shoji, Inc.), 0.2 kg of meat flavor (Takasago International Corporation) was added and mixed to absorb, thereby obtaining an absorbed powdered flavor. This absorbed powdered flavor was forced to free-fall in a feed hopper, horizontally conveyed using a horizontal screw at a screw revolution of 15 rpm to be fed between the compression rollers. The powdered flavor was compacted at a roller compression pressure of 25 N/cm, a roller gap of 0.5 mm and a roller revolution of 15 rpm to obtain a plated matter. The plated matter was cooled with cold air. The cooled plated matter was ground with a rotary grinding machine and evened the size, thereby obtaining a granulated matter.

The granulated meat flavor obtained in Example 12 was used to prepare a meat patty having the following composition.

Ingredients	Composing amount (part weight)
Granulated meat flavor	0.5
Minced meat	650
Onion	200
Bread crumb	40
Egg white	30
Salt	10
Pepper	10

Example 13

To 10.0 kg of sorbitol (Merck Ltd., Japan), 0.2 kg of orange
5 flavor (Takasago International Corporation) was added and mixed
to absorb, thereby obtaining an absorbed powdered flavor. This
absorbed powdered flavor was forced to free-fall in a feed hopper,
horizontally conveyed using a horizontal screw at a screw
revolution of 15 rpm to be fed between the compression rollers.
10 The powdered flavor was compacted at a roller compression
pressure of 25 N/cm, a roller gap of 0.5 mm and a roller revolution
of 15 rpm to obtain a plated matter. The plated matter was cooled
with cold air. The cooled plated matter was ground with a rotary
grinding machine and evened the size, thereby obtaining a
15 granulated matter.

The granulated orange flavor obtained in Example 13 was
used to prepare a powdered orange juice having the following
composition.

Ingredients	Composing amount (part weight)
Granulated orange flavor	0.1

Granulated sugar	7.1
Anhydrous citric acid	0.7
Powdered orange juice	0.1

According to the present invention, granulated flavors having stronger hardness than ever were obtained. Furthermore, the granulated flavors of the invention not only have a great stability of aroma and flavor, capable of retaining the form of the powder and having less fragile property but also have good solubility.

The granulated flavors of the invention retains the flavor within the powder against an external force while releasing the flavor when needed. The form of the granulated flavors of the invention is highly durability to vibration and retains their form upon vibration that may occur during transportation on trucks for distribution of goods containing the granulated flavors and upon physical impact that may occur during production of foods containing the granulated flavors, thereby preventing the commercial products from significantly deteriorating their values. Moreover, the present invention produces a high yield of granulated flavors with desired particle sizes and with good granulation yield, and thus extremely useful for industrial use.

CLAIMS

1. A method for producing a granulated flavor, comprising:
compacting a powdered flavor prepared from raw materials
5 containing a flavor and a carrier with rollers to obtain a
partially melted plated matter;
cooling the plated matter; and
grinding and granulating the cooled plated matter.
- 10 2. A method for producing a granulated flavor, comprising:
forcing a powdered flavor prepared from raw materials
containing a flavor and a carrier to free-fall;
conveying the powdered flavor with a horizontal screw to
obtain a pressed powdered flavor;
15 compacting the pressed powdered flavor with rollers to
obtain a partially melted plated matter;
cooling the plated matter; and
grinding and granulating the cooled plated matter.
- 20 3. A granulated flavor for use in foods and beverages, which
contains a carrier selected from a group consisting of
hydrophilic proteins, maltodextrin, starches, modified
starches, hydrophilic polysaccharides, partially hydrolyzed
proteins, partially decomposed starches and saccharides,
25 wherein the granulated flavor has a moisture content of 10% or
less by weight and a hardness of 1 N/mm²-50 N/mm².

4. A granulated flavor for use in foods and beverages according to claim 3 containing particles having a particle size of 105 μm -2 mm, wherein the proportion of the particles having that particle size is 85% or more by weight.

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5. A food or beverage comprising the granulated flavor for use in foods and beverages of claims 3 or 4.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/JP 03/15239

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A23L1/221 A23C9/16 A23F5/46 A23L1/22

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A23L A23G A23C A23F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6 318 650 B1 (BREITENBACH JOERG ET AL) 20 November 2001 (2001-11-20) column 8, line 49 - line 51; example 3 column 3, line 55 - line 65 column 4, line 37 - column 5, line 2	1-5
X Y	US 5 922 385 A (STIPP GORDON KEITH ET AL) 13 July 1999 (1999-07-13) examples 3,6	1,2,5 3,4
Y	WO 00 25606 A (BENCZEDI DANIEL ;FIRMENICH & CIE (CH); BOUQUERAND PIERRE ETIENNE () 11 May 2000 (2000-05-11) page 18, line 11 - line 25; examples	3,4
	-/--	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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INTERNATIONAL SEARCH REPORT

International Application No

PCT/JP 03/15239

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO 99 32092 A (PALEPU NAGESWARA R ; SMITHKLINE BEECHAM CORP (US); VENKATESH GOPADI) 1 July 1999 (1999-07-01) claims 1,23; examples 2,3,6,8 ---	1-5
Y	US 2001/036503 A1 (BENCZEDI DANIEL ET AL) 1 November 2001 (2001-11-01) paragraphs '0021!', '0032!'; claims 1,3,4,7-10; examples ---	1-5
A	GB 655 592 A (MIJ TOT EXPLOITATIE DER OLIEFA) 25 July 1951 (1951-07-25) claims ---	
A	GB 359 458 A (GILBERT BARKLAY MUSTIN) 19 October 1931 (1931-10-19) ---	
A	WO 85 03414 A (SCM CORP) 15 August 1985 (1985-08-15) -----	

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/JP 03/15239

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 6318650	B1	20-11-2001	DE 19721467 A1 26-11-1998
			AT 204788 T 15-09-2001
			BR 9809860 A 27-06-2000
			CA 2286832 A1 26-11-1998
			CN 1105596 B 16-04-2003
			DE 59801326 D1 04-10-2001
			DK 988106 T3 08-10-2001
			WO 9852684 A1 26-11-1998
			EP 0988106 A1 29-03-2000
			ES 2162707 T3 01-01-2002
			GR 3036715 T3 31-12-2001
			JP 2001527464 T 25-12-2001
US 5922385	A	13-07-1999	US 5741538 A 21-04-1998
WO 0025606	A	11-05-2000	BR 9915007 A 07-08-2001
			CA 2346897 A1 11-05-2000
			EP 1124443 A1 22-08-2001
			WO 0025606 A1 11-05-2000
			JP 2002528601 T 03-09-2002
			US 2001038879 A1 08-11-2001
			CN 1350436 T 22-05-2002
WO 9932092	A	01-07-1999	AU 1931999 A 12-07-1999
			BR 9813808 A 28-05-2002
			CA 2315088 A1 01-07-1999
			CN 1284867 T 21-02-2001
			EP 1047407 A1 02-11-2000
			HU 0100469 A2 28-08-2001
			JP 2001526212 T 18-12-2001
			NO 20003150 A 11-08-2000
			NZ 505123 A 25-07-2003
			PL 341353 A1 09-04-2001
			TR 200001856 T2 21-11-2000
			WO 9932092 A1 01-07-1999
			ZA 9811630 A 21-06-1999
US 2001036503	A1	01-11-2001	BR 0007073 A 31-07-2001
			CA 2346948 A1 15-03-2001
			EP 1124442 A1 22-08-2001
			WO 0117372 A1 15-03-2001
GB 655592	A	25-07-1951	NONE
GB 359458	A	19-10-1931	NONE
WO 8503414	A	15-08-1985	CA 1231934 A1 26-01-1988
			EP 0169893 A1 05-02-1986
			JP 61501078 T 29-05-1986
			WO 8503414 A1 15-08-1985
			US 4689235 A 25-08-1987